ISSUE : 1120/11

HIDEN ANALYTICAL

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Mass Spectrometers for Plasma Characterisation

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Christopher Muratore ⋮ University of Dayton

CF_x films synthesized by HIPIMS sputtering of carbon
Dr. Susann Schmidt ⋮ Linköping University

Investigation of Solar Energy Materials
D. M. Meysing ⋮ Colorado School of Mines

CH_2F^+ and CHF_2 ion density measurements in argon or krypton-diluted CH_2F_2 plasmas
Yusuke Kondo, Kenji Ishikawa, Toshiya Hayashi, and Masaru Hori ⋮ Nagoya University

Time resolved Langmuir probe diagnostics for ECR plasma research
O.D. Cortázar ⋮ ESS Bilbao Consortium

Related Products:

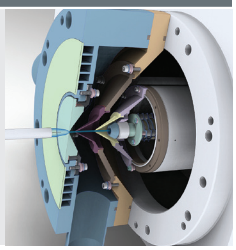
EQP - Plasma Sampling Mass Spectrometer

ESPion - for Measurement of Plasma Properties

HPR-60 - Molecular Beam Mass Spectrometer

In the press:

Mass spectrometric measurement of plasma and flame chemistry



A very big thank you to all who have contributed:

COLORADO SCHOOL OF MINES — EARTH • ENERGY • ENVIRONMENT · NAGOYA UNIVERSITY · ESS bilbao · UNIVERSITY of DAYTON · GT · AFRL · Linköpings Universitet

Investigation of Solar Energy Materials

Our Reference: AP-EQP-0003

PROJECT SUMMARY BY:

COLORADO SCHOOL OF MINES
EARTH • ENERGY • ENVIRONMENT

D.M. Meysing
Colorado School of Mines
1500 Illinois St., Golden, CO 80401 USA

PAPER REFERENCE:

1) D.M. Meysing, J.M. Burst, W.L. Rance, M.O. Reese, T.M. Barnes, T.A. Gessert, C.A. Wolden, The influence of cadmium sulfide and contact annealing configuration on the properties of high-performance cadmium stannate, *Solar Energy Materials and Solar Cells* **117** (2013) 300–305.

2) W.L. Rance, J.M. Burst, D.M. Meysing, C.A. Wolden, M.O. Reese, T.A. Gessert, W.K. Metzger, S. Garner, P. Cimo, T.M. Barnes, 14%-efficient flexible CdTe solar cells on ultra-thin glass substrates, *Applied Physics Letters* **104**, (2014) 143903.

HIDEN PRODUCT:

EQP

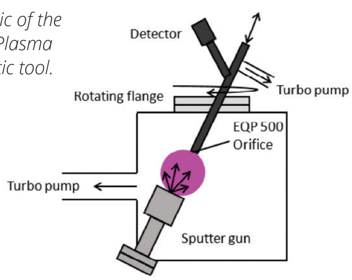
Daniel Meysing is a Ph.D. candidate in the Chemical and Biological Engineering Department at the Colorado School of Mines in Golden, Colorado. He is advised by Prof. Colin Wolden and Adjunct Prof. Teresa Barnes from CSM. Here, he describes his research.

Research

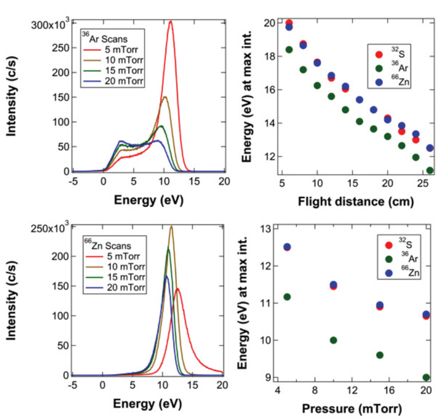
Our group began working with the Hiden EQP 500 about one year ago. We are investigating the chemistry and energetics in the radio frequency magnetron sputtering of II-VI compounds used in thin-film solar cells. Sputter deposition of these materials is often treated as a "black box" in which we control the input parameters (sputtering power, pressure, ambient composition) and determine the film properties (optical transmission, conductivity, composition) without detailed knowledge of the process itself. Our goal is to develop a better understanding of some of the complex mechanisms in sputtering that affect optoelectronic properties in thin films.

The sputtering chamber, termed the Sputter-Plasma Diagnostic (SPD) tool, accommodates up to six sputtering guns in sputter-up configuration. A rotating flange, on which the Hiden EQP is mounted, and a z-axis motor allow the Hiden EQP 500 analyser to be placed normal to or off-axis to the sputtering target and moved closer to or further away from the target. The EQP is differentially pumped and can be fitted with different diameter orifices so that it can be used over a range of different pressure regimes. We use a 100-μm aperture in the current work to enable analysis at typical sputtering pressures of ~10 mTorr.

Schematic of the Sputter-Plasma Diagnostic tool.



Some of our initial work has focused on plasma-generated ion energy distributions (IED) during radio frequency magnetron sputtering of ZnS in a pure Ar ambient. We have probed parameters such as the power, pressure, and flight distance of Ar, Zn, and S ions. Ar ions have distinctly different IEDs compared to Zn and S. The Ar IED has a low-energy shoulder caused by reflection at the target and Penning ionization reactions, whereas Zn and S IEDs have a single dominant peak and often a small high-energy shoulder. In all cases, the Zn and S peaks are ~1.5 eV higher in energy than the Ar peak. The effect of flight distance was investigated by varying the displacement between the target and the EQP orifice. As expected, additional collisions caused the arrival energy of ions to decrease with increasing flight distance. In a similar manner, the ion energy decreased with increasing pressure.



Zinc sulfide ion energy distribution scans. (Top left) ^{36}Ar profiles as a function of pressure; (Bottom left) ^{66}Zn profiles as a function of pressure; (Top right) ^{32}S, ^{36}Ar, and ^{66}Zn IED peak positions as a function of flight distance; (Bottom right) ^{32}S, ^{36}Ar, and ^{66}Zn IED peak positions as a function of pressure.

Future Research

We plan to study oxygen incorporation during reactive sputtering of ZnS and CdS in an O_2/Ar ambient. Oxygen is known to incorporate via substitution and/or reaction with sulfur.

Our poster, entitled "Energy-resolved quadrupole mass spectrometry in II^B-VI^A sputtering investigations," won third place at the 2014 Rocky Mountain American Vacuum Society Symposium held in Denver, Colorado.

Hydrofluorocarbon ion density of argon- or krypton-diluted CH_2F_2 plasmas: generation of CH_2F_+ and CHF_{2+} by dissociative ionization in charge exchange collisions

Quadrupole mass-spectroscopic analysis – in rare gas (M) diluted CH_2F_2 plasma – has revealed selective formation of CH_2F^+ ion for Ar dilution and CHF_2^+ ion for Kr dilution. Ion densities of CH_2F^+ and CHF_2^+ were determined by dissociative ionization pathways in channels of charge exchange collisions, *i.e.*, $CH_2F_2 + M^+ \rightarrow CH_2F^+ + F\cdot + M^*$ and $CHF_2^+ + H\cdot + M^*$ in CH_2F_2 plasmas. In Ar-diluted plasmas, CH_2F^+ ions predominated due to dissociative ionization between Ar^+ (*ca.* 15.8 eV) and C-F appearance energy (*ca.* 16 eV) to form CH_2F^+. In contrast, for Kr-diluted plasmas, C-H appearance energy (*ca.* 13.8 eV) predominated to produce a larger amount of CHF_2^+ ions due to a similar channel for charge exchange collisions between Kr^+ (*ca.* 14 eV) and CH_2F_2. In accordance with the analytic results, the addition of Ar and Kr gas to CH_2F_2 plasmas provided control over the fraction of CH_2F^+ and CHF_2^+ ion densities.

Hydrofluorocarbons have H atoms in place of F atoms in fluorocarbon gases. Dissociation reactions involving C−H and C−F bonds are of interest for controlling the density of reactive species: F atoms, produced by dissociation of the C−F bond, are a main etchant for Si, while H atoms, produced by dissociation of the C−H bond, promote the deposition of polymers on a substrate surface. For processing accuracy, a balance of species for etching and deposition is believed to be important and to be closely related the dissociation processes in gas-phase. However, the variety and densities of the ions and radicals generated in hydrofluorocarbon plasmas have not been fully elucidated.

The experiments was performed with a dual frequency capacitively coupled plasma (CCP) etching reactor, installed a quadrupole mass spectrometer (QMS; Hiden Analytical, EQP) at the chamber wall. A 100-µm diameter aperture was installed in the QMS entrance. A mixture of Ar or Kr gas with CH_2F_2 gas was introduced into a chamber, and plasmas were sustained by applying the very high frequency (VHF) power to the electrode.

Positive ion mass spectrometric measurements revealed that the dominant positive ions were CH_2F^+ and CHF_2^+. In the ionization pathway generated for CH_2F^+ and CHF_2^+ ions, two channels are involved: CH_2F^+ through C-F bond dissociation or through C-H bond dissociation. The reaction schemes for the dissociative reactions in electron collisions are given by $CH_2F_2 + e^- \rightarrow CH_2F^+ + F + 2e^-$ (threshold at 15.8 eV), and $\rightarrow CHF_2^+ + H + 2e^-$ (13.8 eV). The counter fragments of charge-neutral H and F atoms were generated simultaneously through these dissociation mechanisms. A larger ion density for CH_2F^+ in the Ar-diluted plasma, other dissociation processes but the electron collisions need to be considered. Charge exchange collisions between rare gas ions and CH_2F_2 molecules occurred, because the appearance energies were located close to that for Ar (16 eV) and Kr (14 eV).

We concentrate our continuous study in elucidation of the dissociative reactions in plasma through the gas-phase diagnostics utilized the mass-spectrometric measurements.

Our Reference: **AP-EQP-0005**

PROJECT SUMMARY BY:

NAGOYA UNIVERSITY

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Nagoya University, Furo-cho, Chikusa, Nagoya 464-8603, JAPAN

PAPER REFERENCE:

Yusuke Kondo *et al.,* (2015) "Hydrofluorocarbon ion density of argon- or krypton-diluted CH_2F_2 plasmas: generation of CH_2F^+ and CHF_2^+ by dissociative-ionization in charge exchange collisions" *J. Phys. D: Appl. Phys.* **48** 045202. doi:10.1088/0022-3727/48/4/045202

HIDEN PRODUCT:

EQP Mass & Energy Analyser

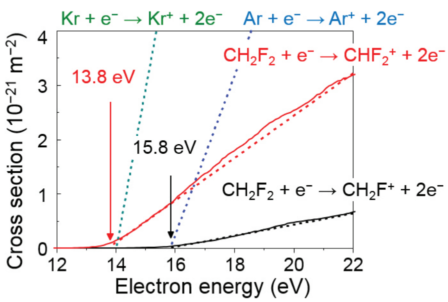


FIGURE 1: *Cross section for dissociative ionization for a CH_2F_2 molecule*

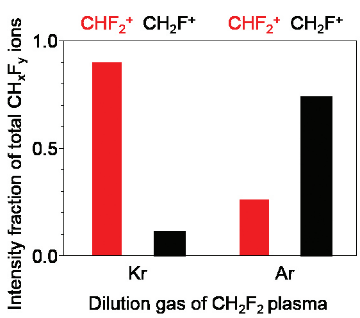
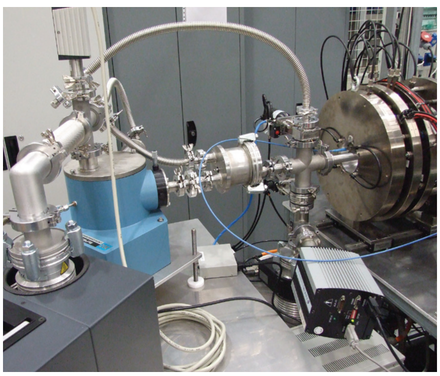


FIGURE 2: *Individual CH_2F^+ and CHF_2^+ ion fraction on total CH_xF_y + ion density at Kr- and Ar-diluted CH_2F_2 plasma*

ECR plasma research project for ion sources at ESS Bilbao: Breakdown studies by time resolved Langmuir probe diagnostic

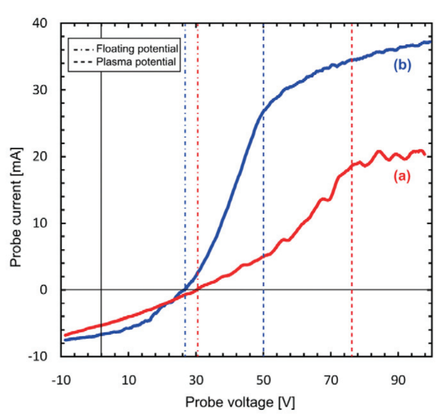
Transient effects in pulsed electron cyclotron resonance (ECR) plasmas are of high interest for applications, e.g. particle accelerators and plasma processing industry. A research project on this subject is being conducted at ESS Bilbao (www.essbilbao.org) in Spain as a part of a bigger program related to accelerator technology and spallation neutron generation. T.I.P.S. (Test bench for Ion-sources Plasma Studies) is an ECR plasma generator driven by a 2.45 GHz and 3 kW adjustable power magnetron that has been developed to be used as a flexible facility to conduct a research program focused on the plasma physics associated to ECR ion sources performance.



A view of the experiment with time resolved Langmuir probe and VUV spectroscopy diagnostics.

The diagnostics setup utilizes several complimentary techniques i.e. directional couplers for incident and reflected power measurement, vacuum-ultraviolet (VUV) spectrometer and Langmuir probe. The studies are intended to broaden the understanding of the plasma breakdown dynamics of ECR plasmas. In particular, it reveals similarities of the plasma breakdown transient effects observed earlier with high frequency ECR ion sources intended for the production of multi-charged ions and 2.45 GHz microwave discharges often used for the production of high current mono-charged ion beams.

The Langmuir probe system is used for acquiring I-V curves permitting to estimate plasma electron temperature and density. The probe tip is made of 6 mm long, 0.5 mm diameter tungsten wire. Transient effects can be studied by synchronizing the probe with the VUV-emission measurement via a delay generator and building the I-V curve from the data obtained over consecutive pulses. ***The Langmuir probe driver circuit (ESPion) is made by Hiden Analytical LTD*** and it acquires a single I-V point in 62.5 ns and rearms itself in another 14.6 μs required for data handling. The jitter between the timing signals is kept below 200 ns. Each point of the I-V curve is an average of the probe currents acquired over 100 consecutive pulses at fixed probe voltage. Altogether it takes several minutes to acquire a complete I-V curve corresponding to a single temporal data point. The electron temperature T_e is estimated from the I-V curve (below) assuming Maxwellian electron energy distribution function (EEDF). A temperature peak reaching almost 20 eV is observed in coincidence with a drastic reduction of reflected power during microwave coupling process. Such peak is followed by a decreasing behaviour that reaches about 5 eV as final steady state temperature, remaining practically constant during flat top microwave pulse. Electron density reaches stable values about 1.5×10^{16} m^{-3} at the time when temperature peak is produced, fact that suggests this process as deeply associated to plasma evolution during breakdown. This is also confirmed by time resolved VUV spectroscopy measurements where similar peaking behaviour observations are recorded for Lyman-alpha and Lyman band emission.



ESPion Langmuir Probe I-V curves. Curve (a) typical data corresponding to plasma breakdown, Curve (b) typical data taken during steady-state plasma conditions (15 and 60 μs after an incoming microwave pulse)

Our Reference: **AP-ESP-0001**

PROJECT SUMMARY BY:

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Paseo Landabarri 2,
48940 – Leioa, Vizcaya - SPAIN

PAPER REFERENCE:

O. D. Cortázar, J. Komppula, O. Tarvainen, A. Megía-Macías, A. Vizcaíno-de-Julián and H. Koivisto (2013) "Experimental study of hydrogen plasma breakdown in a 2.45 GHz microwave discharge" *Plasma Sources Sci. Technol.* **22**, 015026 (9pp)

HIDEN PRODUCT:

ESPion Advanced Langmuir Probe

Nanoparticle decoration of carbon nanotubes by sputtering

Nanoparticle-decorated carbon nanotubes (CNTs) are effective chemical and biological sensors, surfaces for heterogeneous catalysis, photovoltaics, and conformal thermal interface materials for electronics. The particle morphology on the CNT sidewalls strongly affects the properties and performance of metal-nanotube hybrids for such applications. Often nanoparticles are deposited by electrochemical methods, which generally require time consuming treatments with strong acid for surface defect production, which can result in a compromise of the intrinsic mechanical or transport properties of the CNTs, inhibiting their multi-functionality. We have examined physical vapor deposition techniques as scalable alternatives to electrochemical treatment for in situ growth of metal nanoparticles on the sidewalls of multi-wall carbon nanotubes (MWCNTs). Vapor phase growth of gold, nickel and titanium metal nanoparticles on multi-wall carbon nanotube (MWCNT) bucky paper was investigated. The size and distribution of nanoparticles was dependent on the intrinsic binding energy of the elemental metals, where metals with larger cohesive energies exhibited a higher nanoparticle density and smaller particle diameters.
Particle diameters for any metal could be altered to mimic that of metals with different binding energies by in situ modification of the MWCNT surfaces by energetic metal ions (characterized with a Hiden EQP 1000 as shown in Figure 1) during their growth, where removal of a carbon atom from a MWCNT surface requires incident ions kinetic energies > 5-7 eV. Control of the ariel density, diameter and morphology of metal nanoparticles grown on as-received and annealed multi-walled carbon nanotube sidewalls by sputtering was demonstrated for gold, nickel and titanium.

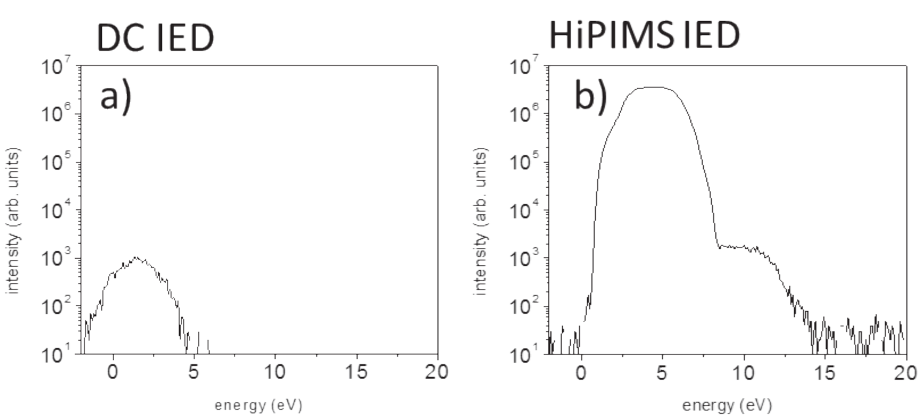

FIGURE 1: *Au^+ ion energy distributions measured for (a) dc and (b) HiPIMS processing conditions.*

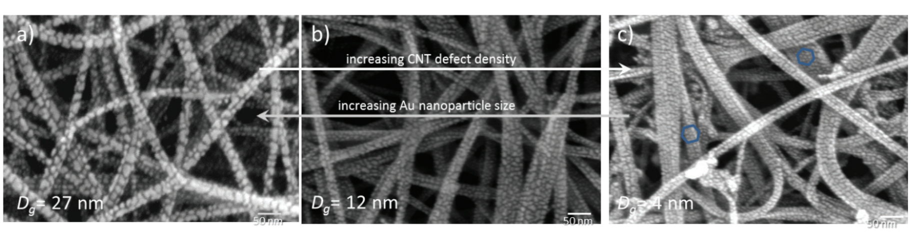

FIGURE 2: *Scanning electron micrographs and accompanying Raman spectra for (a) annealed buckypaper with dc nanoparticle growth, (b) annealed buckypaper with HiPIMS growth, and (c) as-received buckypaper with HiPIMS growth.*

Our Reference: **AP0599**

PROJECT SUMMARY BY:

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PAPER REFERENCE:

C. Muratore, A.N. Reed, J.E. Bultman, S. Ganguli, B.A. Cola, A.A. Voevodin (2013) "Nanoparticle decoration of carbon nanotubes by sputtering" *Carbon* **57** 274–281

HIDEN PRODUCT:

EQP 1000

A range of average gold particle diameters from approximately 5-30 nm could be produced depending on the intrinsic sputter process parameters (especially metal ion flux and kinetic energy) and defect density of the MWCNT surfaces which could also be controlled by annealing prior to sputtering (Figure 2). Particle characteristics could also be altered with temperature, total incident ion flux during processing, or by initial CNT diameters. The diameter of the MWCNTs had a significant influence on the geometry of the nanoparticles.

Particles were elongated along the nanotube axis for tube diameters <30 nm. Remarkably strong alignment of the particles along the nanotube axis was observed, especially for MWCNTs with higher defect densities. The PVD process used to grow the nanoparticles is easily scalable to large arrays of CNTs, and will be explored for adding multi-functionality to CNT-based nanocomposites.

CF_x films synthesized by reactive high power impulse magnetron sputtering of carbon in argon/tetrafluoromethane (Ar/CF_4) and argon/octafluorocyclobutane ($Ar/c\text{-}C_4F_8$) atmosphere

The synthesis of amorphous CF_x thin films by reactive high power impulse magnetron sputtering (rHiPIMS) was demonstrated in tetrafluoromethane and octafluoro-cyclobutane atmospheres. All depositions and the plasma characterization by positive ion mass spectrometry (Hiden EQP 1000, Hiden Analytical Ltd., UK) were carried out in an industrial coater (CC 800/9ML, CemeCon AG, Germany) utilizing a substrate temperature of 110ºC and a process pressure of 400 mPa. The CF_x film composition as measured by elastic recoil detection analysis was varied in the range of $0.15 < x < 0.35$ by regulating the partial pressure of the F-containing gases from 0 mPa to 110 mPa. Results from process and plasma characterization were related to *ab initio* calculations and CF_x thin film properties. Our DFT calculations predicted CF, CF_2, CF_3, as well as C_2 and F to be the most important precursor species for the film growth in Ar/CF_4 mixtures. For carbon discharges in Ar/C_4F_8 mixtures, additionally, C_2F_2 was predicted to play a significant role. Results obtained from time averaged positive ion mass spectrometry agree well with our theoretical calculations. Here, Ar^+, C^+, CF^+, CF_2^+, CF_3^+, as well as F^+ were found to be abundant cations (cf. figure 1 a) and b)). The characterization of the rHiPIMS processes revealed moreover two deposition regimes depending on the partial pressure of the F-containing reactive gas; as the partial pressure rises above 42 mPa in Ar/CF_4 plasmas an ionization cascade progresses, resulting in a rising peak target current and an increased formation of CF_4 fragments, especially CF_3. In C_4F_8 plasmas the ionization cascade onset was observed for partial pressures above 11 mPa, accompanied with an increased production of CF. These two regimes are mirrored in the thin film properties, particularly in their chemical bond structure, hardness and elastic modulus. The mechanical response of the CF_x films can also be related to abundant precursor ions in the plasma; in C_4F_8 discharges next to Ar^+ and C^+, CF^+ species are most abundant. CF^+ and CF possess three dangling bonds, and are consequently reactive as well as able to build strong cross links in the carbon matrix. Therefore, CF_x films deposited in C_4F_8 show an elevated hardness over a wider range of incorporated F (cf. figure 2). In contrast, for Ar /CF_4 discharges CF_3^+ was determined as the species of highest abundance. CF_3^+ and its neutral counterpart have one dangling bond and thus does not significantly contribute to cross linking of the growing film. This is mirrored in a rapid decrease of the hardness as the F content in the films increases. Therefore, it can be concluded that the use of C_4F_8 has advantages with regards to the controllability of the film properties, while CF_4 covers a wide range of the applicable process window - thin film deposition and etching. Additionally, the dissociation of CF_4 into primarily CF_3 and F can be utilized for surface treatments and surface termination leading to low surface energies. Consequently, the rHiPIMS processes in C_4F_8 or CF_4 present a versatile tool for the further functionalization of carbon and carbon based thin films as well as surfaces.

Our Reference: **AP0686**

PROJECT SUMMARY BY:

Dr. Susann Schmidt
IFM, Thin Film Physics, Division, Linköping University, S-581 83 Linköping, Sweden

PAPER REFERENCE:

S Schmidt et al. (2013) "Reactive high power impulse magnetron sputtering of CF_x thin films in mixed Ar/CF_4 and Ar/C_4F_8 discharges" *Thin Solid Films* **542** (2), 21-30

HIDEN PRODUCT:

EQP 1000

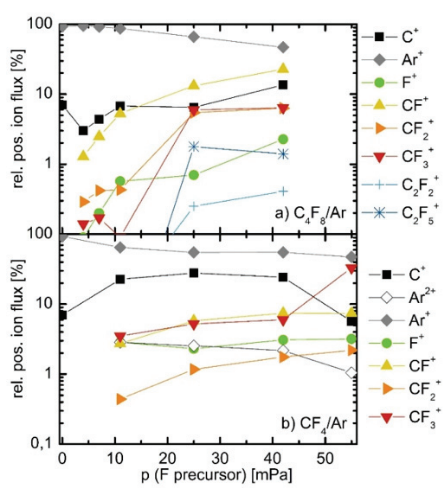


FIGURE 1 A) AND B): *Relative ion fluxes as a function of the reactive gas partial pressure for discharges in a) Ar/C_4F_8 and b) Ar/CF_4 extracted from time averaged IEDFs of corresponding processes.*

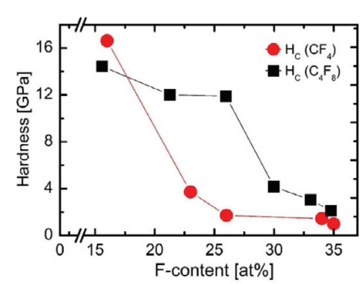


FIGURE 2: *Hardness over the fluorine content for CF_x films grown in Ar/C_4F_8 (black squares) and Ar/CF_4 (red circles).*

Related Products:

EQP – Plasma Sampling Mass Spectrometer

The Hiden EQP is a combined mass/energy analyser for the analysis of positive AND negative ions, neutrals, and radicals from plasma processes:

- Analysis of positive ions, negative ions, neutral radicals and neutrals
- Etching/Deposition Studies
- Ion Implantation/Laser Ablation
- Residual Gas Analysis/Leak Detection
- Plasma electrode coupling - follow electrode conditions during operation
- Analysis through a viewport, grounded electrode, driven electrode

ESPion – for Measurement of Plasma Properties

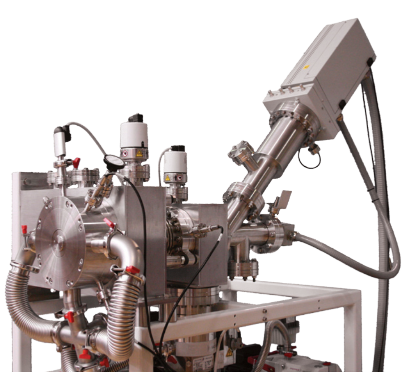
The ESPion advanced Langmuir probe for rapid, reliable and accurate plasma diagnostics for industry and academia:

- Etching / Deposition / Cleaning Plasma Processes
- Pulsed plasma operation
- Ion density (Ni & Gi)
- Electron retardation (Te & EEDF)
- Electron density (Ne)
- Plasma Potential
- Debye Length, floating potential
- Ion flux

HPR-60 – Molecular Beam Mass Spectrometer

The Hiden HPR-60 Molecular Beam Sampling Mass Spectrometer is a compact gas analysis system for analysis of neutrals, radicals and ions:

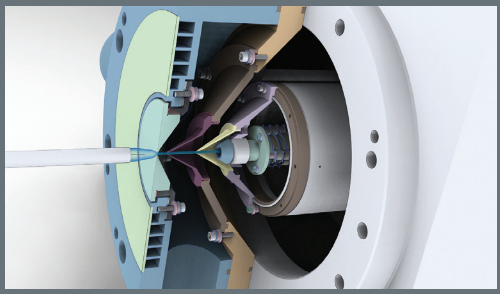
- Reaction Kinetics
- Plasma Diagnostics
- Combustion Studies – Flame Ionisation Analysis
- Catalysis Studies
- CVD/MOCVD – Diamond Growth Studies
- Flash Desorption Studies
- Atmospheric Glow Discharge Analysis
- Cluster Analysis



In the press:

OUR REFERENCE: HAPR0123

Mass spectrometric measurement of plasma and flame chemistry



Hiden HPR-60 Plasma-Flame Diagnostic

The mass spectrometric analysis of the ionised and chemically unstable reactive species generated in flame and high-pressure plasma environments requires their rapid transit through vacuum through to the mass spectrometer with minimal interaction.

The Hiden HPR-60 system is a research tool designed specifically for these analyses at process pressures from sub-atmospheric through to 10 bar. Multiple ultra high vacuum (UHV) stages with independent turbomolecular vacuum pumping are separated by coned diaphragms each with a central orifice, all precisely aligned to enable the unhindered transmission of the sampled beam through to the mass spectrometer probe for analysis.

The mass spectrometer is the Hiden EQP Mass/Energy analyser measuring mass and energy of both positive and negative ions, with the on-board electron bombardment ion source providing analysis of neutrals. A mechanical beam chopper enhances detection levels for neutrals by modulating the beam, data acquisition then being gated to enable direct comparison of beam-on/beam off intensities for subtraction of background elements.

Hiden HPR-60 Plasma-Flame Diagnostic Systems provide detailed analysis of plasma and flame chemistry together with reaction kinetics and confirmation of gas-phase intermediate species. A custom design service is available to assist with specific user interface requirements.

For further information on these or any other Hiden Analytical products please contact Hiden Analytical at **info@hiden.co.uk** or visit the main website at **www.HidenAnalytical.com**

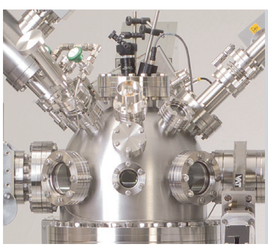
If you would like to submit a project summary for consideration in our next Newsletter, please email a brief summary (*approx. 500 words*) and corresponding images to **marketing@hiden.co.uk**

Hiden**APPLICATIONS**

Hiden's quadrupole mass spectrometer systems address a broad application range in:

GAS ANALYSIS

- dynamic measurement of reaction gas streams
- catalysis and thermal analysis
- molecular beam studies
- dissolved species probes
- fermentation, environmental and ecological studies

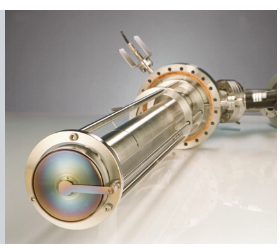


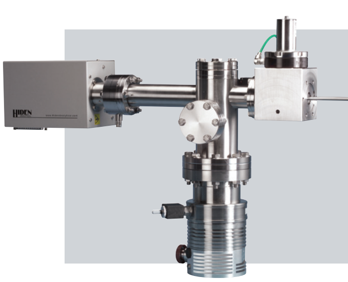
SURFACE ANALYSIS

- UHV TPD
- SIMS
- end point detection in ion beam etch
- elemental imaging – 3D mapping

PLASMA DIAGNOSTICS

- plasma source characterisation
- etch and deposition process reaction kinetic studies
- analysis of neutral and radical species





VACUUM ANALYSIS

- partial pressure measurement and control of process gases
- reactive sputter process control
- vacuum diagnostics
- vacuum coating process monitoring

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